

Micro-Scale Mechanical Testing of Non-Woven Carbon Nanotube Sheets and Yarns

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ABSTRACT

Non-woven carbon nanotube (CNT) sheets and yarns were tested using a novel micro-scale mechanical testing system. CNT sheets were observed to delaminate during uniaxial testing using an adhesive gripping method, resulting from a higher proportion of load bearing in the outer sheets versus internal sheets and an apparently low interlaminar shear strength. In response to this, a new spool-grip method was used to alleviate non-uniform through-thickness stresses, circumvent premature delamination, and allow the sheet material to sustain a 72% increase in measured tensile strength. Furthermore, tension tests of CNT yarns showed that the yarn-structure was approximately 7 times stronger than the sheet structure, owing to a higher degree of CNT alignment in the test direction.

INTRODUCTION

Nano-structured materials show great promise for future space technologies and applications such as high strength and lightweight carbon nanotube (CNT) based structural materials. For example, the discovery that CNTs have a specific strength about 200 times greater than steel yet are 5 times less dense [1] may enable a new realm of structural space materials that can allow the deployment of higher capacity payloads in future space missions. In order to measure the strength of these types of nano-structured materials, early experiments required the use of MEMS and NEMS-based testing devices [1]. However, this scale of measurement device is not practical for newly emerging, larger scale, mass-produced nano-materials. Similarly, some materials are too small in size (typically one or more characteristic lengths below 1 mm) or too delicate and brittle (like thin films or coatings that are prone to cracking) to mount and test in a conventional testing machine. Additionally, the available quantity of material to be tested may be limited or scarce. For example, if an engineer were to test the effect of space-exposure on the mechanical

strength of a small quantity of insulation layers retrieved from the Hubble Space Telescope, it would be advantageous to minimize the use of such a limited supply of material [2]. As a result, NASA Goddard Space Flight Center has developed a system that allows for the testing of materials with micron-scale features (such as micron-thick sheets and micron-diameter fibers) with in-situ image-based measurement capabilities.

For this research, the mechanical behavior of two sets of CNT-based materials were experimentally measured: non-woven carbon nanotube (CNT) sheets (approximately 50 μm thick) and CNT yarns consisting of bundled CNT fibers spun into a rope-like material (approximate 50 μm diameter, see Fig. 1). The micro-mechanical testing system is described, along with a novel gripping technique and the associated experimental procedure to measure the mechanical behavior of the nano-scale materials. A comparison is made between the strength of the sheet and yarn materials, and the implications of microscale metrology and testing methods on the characterized behavior of nano-based and micro-sized materials are discussed.

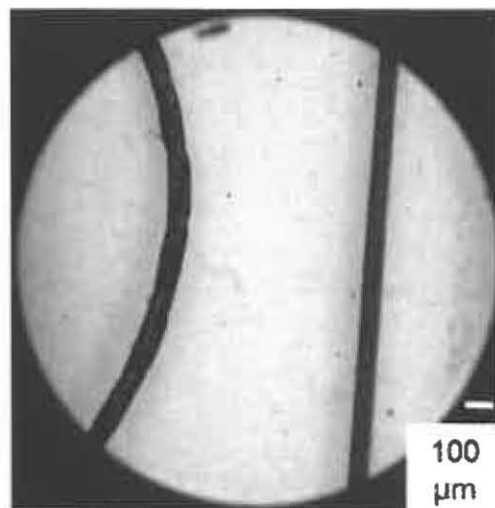


Fig. 1: CNT yarn (left) and human hair (right)

EXPERIMENTAL SETUP

A. MICRO-MECHANICAL TESTING SYSTEM

The micro-scale mechanical testing system is comprised of a 6-axis motion stage to provide actuation (14 mm maximum travel distance, 1.2 mm/min maximum linear velocity, 65 nm spatial resolution), a linear air-bearing to reduce off-axis loading of the specimen, a tension/compression load cell to measure the resulting pulling force (10 gram, 100 gram, 250 gram, and 900 gram capacities), and an optical microscope combined with a CCD camera to record images of the material during deformation (11.25x maximum magnification). Specimens are mounted in-line between the actuating motion stage and the load cell atop a pair of specimen grip platforms. Actuator motion as well as sensor data and image acquisition are all controlled using a fully integrated computer control program. An image of the system is shown in Fig. 2.

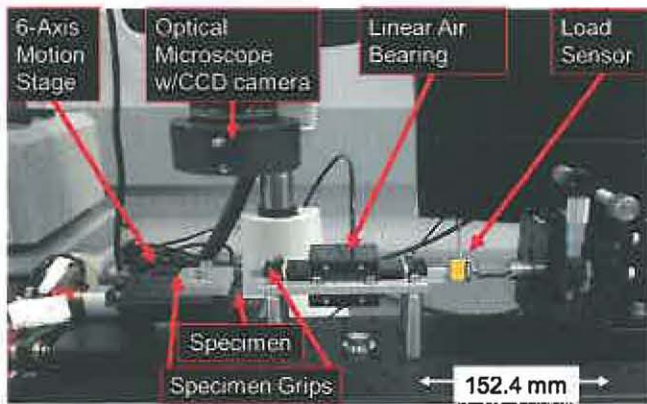


Fig. 2: Micro-Scale Mechanical Testing System

B. SPECIMEN PREPARATION

Tension specimens of the 40-50 μm thick sheet material were sectioned to an approximate 250 μm width using a diamond saw blade to minimize machining edge defects. The sheet materials were observed to have an inherent orthotropic orientation due to the material fabrication process, and therefore for this study only one orientation was consistently used for characterization. Sheet specimens were visually inspected under an optical microscope prior to testing to ensure minimal machining defects and to measure specimen geometry. Yarn materials (with diameters ranging between 40-60 μm) were carefully sectioned to size using a sharp razor blade. All materials were sectioned to an approximate 10 mm total length, resulting in a 4 mm specimen gage length during testing.

C. SPOOL-GRIP FIXTURES

Due to the delicacy of the specimens, traditional clamping methods for gripping were not possible. It was also determined that the use of an ultraviolet (UV)-curable adhesive to mount the tension specimens to flat metal pads

was not ideal for the tested materials, as delamination would occur during experiments. As a result, a new gripping fixture was designed, fabricated, and employed for tension tests. The 'spool-grip' fixture allows the user to wrap thin sheets, fibers, or yarns of material around two spool-like pins. The fixture consists of a set of stainless steel pins, approximately 1.6 mm in diameter, with a 500 μm wide and 500 μm deep notch machined longitudinally along the surface each pin (using electric discharge machining). Each pin is inserted into a steel fixture which is secured to either side of the specimen grip platforms of the testing machine. A pair of set screws are used to prevent rotation of the spool pins during the tension test. A schematic and representative image of the spool-grip fixture is shown in Fig. 3.

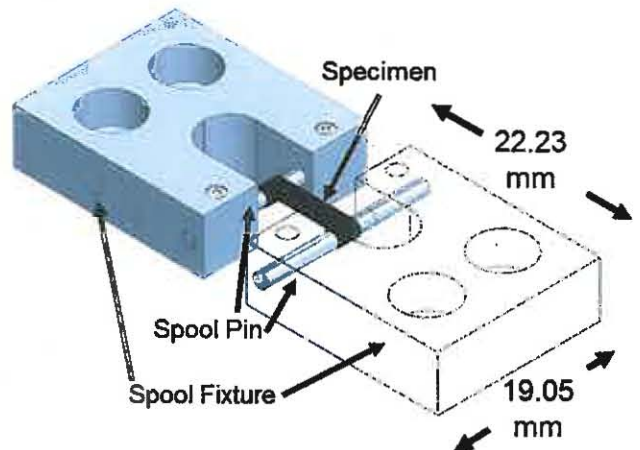
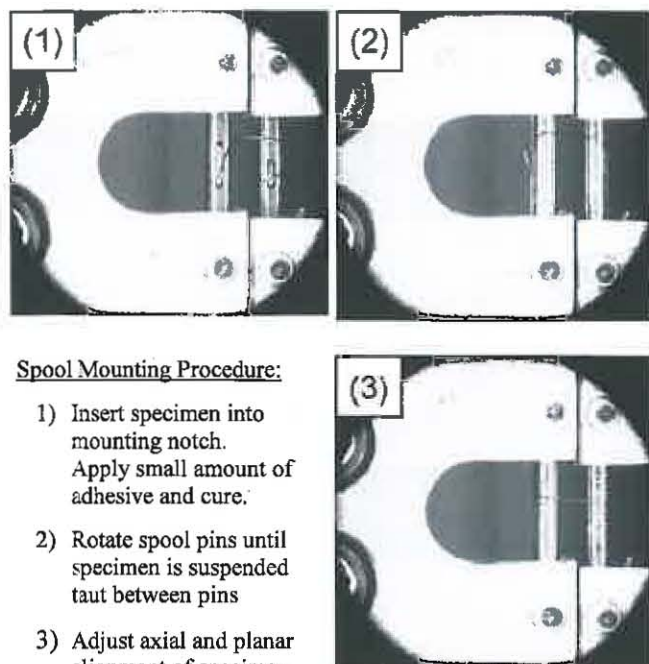


Fig. 3: Spool-grip fixture for micro-sized specimens

D. SPOOL-GRIP MOUNTING PROCEDURE

To mount specimens using the spool-grip method, each end of the specimen is first inserted into the mounting notch of each pin. A small amount of adhesive can be used to hold the ends of the specimen inside each mounting notch. Next, each spool-pin is slowly rotated in opposing directions until the specimen is suspended taut between each pin (note: during this step it is important to monitor the load across the specimen and ensure the specimen is not experiencing any appreciable pretension). Finally, the axial and planar alignment of the specimen can be adjusted using the motion

stage to ensure proper uniaxial alignment during testing. Figure 4 shows an example of a typical mounting procedure for a micro-sized specimen (strand of human hair, approximately 50 μm in diameter and similar size compared to the CNT yarns tested in this research).



Spool Mounting Procedure:

- 1) Insert specimen into mounting notch. Apply small amount of adhesive and cure.
- 2) Rotate spool pins until specimen is suspended taut between pins
- 3) Adjust axial and planar alignment of specimen. Begin test.

Fig. 4: Mounting procedure for spool-grip method. Images show mounting of human hair specimen (similar diameter compared to CNT yarns)

E. TESTING PROCEDURE

Uniaxial tension tests were conducted at a constant displacement rate of 0.1 mm/min, yielding a strain rate of approximately 10^{-4} s^{-1} . During the tests the displacement of the specimen grips, load required to deform the specimens, and images of the deforming specimens were all recorded using an integrated data acquisition program in LabVIEW. Tests were conducted using two different mounting configurations for the CNT sheets: an adhesive grip method—which was found to cause bonding issues, and the spool grip method. The mounting procedure for the adhesive grip method consists of carefully placing each end of the specimen directly on the testing machine grip platforms, applying a small amount of UV-curable adhesive to the grip sections of the specimens, and curing the adhesive for approximately 30 seconds under a high intensity UV light. Only the spool grip method was used for the CNT yarns due to early signs of specimen debonding during preliminary trials using the adhesive grip method. Three experimental trials were conducted for each testing condition using the CNT sheets, while only two trial results are shown for the yarns.

F. STRAIN MEASUREMENT

For all experiments, engineering strain was first estimated by dividing grip displacement by the initial specimen gage length. However, a more accurate method of directly measuring the strain of the sheet materials was employed using digital image correlation (DIC) software (Icasoft, from INSA-Lyon). To achieve successful image-based strain measurement, a random speckle pattern was produced on the surface of the specimens prior to testing using a fine dusting of alumina powder. After experiments were conducted, the captured images were post-processed using DIC software to calculate full-field displacement and strain fields. A virtual linear extensometer measurement feature was used to calculate the uniaxial strain of the specimens. An example of images taken during a tension test of a CNT sheet specimen, as well as full-field DIC strain measurements, is shown in Fig. 5.

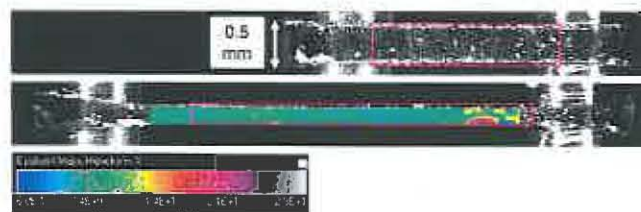


Fig. 5: CNT sheet specimen before uniaxial tension (top) and during tension test just prior to fracture (bottom). DIC-measured strain field is displayed on deformed image

It is important to note that the deformation of the sheet specimens often caused the surface morphology of the specimens to drastically change from their original, undeformed morphology. This is significant for DIC strain measurement as subsequent displacement and strain computations are determined with respect to the initial undeformed image. As a result, a modification was made to the DIC analysis procedure in which each set of experimental images was divided into sets of 200 consecutive images and strains were computed within each image set, taking the first frame in each set as the strain reference image. The total engineering strain was then computed by taking the sum of each set of incrementally measured strains. For example, Fig. 5 shows the measured strain field at fracture with respect to the 1000th captured image and not the first; therefore, the displayed average strain is about 8% at fracture compared to total strain-at-fracture of 36%. DIC analysis was not possible for the CNT yarns due to a difficulty in preparing a proper speckle pattern for sufficient image analysis. Therefore, strain results for the CNT yarns are presented using estimations from grip displacement.

RESULTS

A. CNT SHEETS

The uniaxial tension behavior of the CNT sheets using the adhesive grip method is shown in Fig. 6. The measured tensile strength was consistent through all trials, resulting in an average tensile strength of 20.7 ± 0.5 MPa. When grip displacement is used to compute strain, it can be seen that the failure strains of the sheet materials seem to range from 54-173%. However, the DIC method of strain measurement shows that the actual, directly measured uniaxial strain experienced by the sheet is far less (failure at 21% compared to 54%). The reason for this will be discussed in the next section. This discrepancy is also apparent when computing the Young's Modulus of the material as the DIC method presents a stiffer elastic response of the sheet with a Modulus of 353 MPa, compared to about 200 MPa using the grip displacement method.

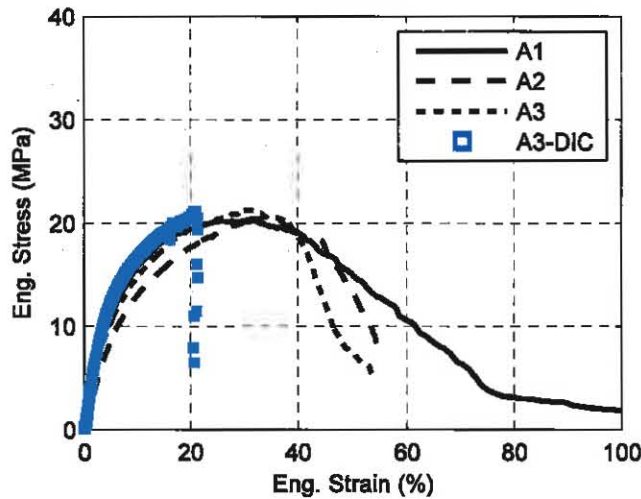


Fig. 6: Uniaxial tension test results for CNT sheets using adhesive-grip method

It was visually observed during experiments using the adhesive-grip method that the sheet specimens demonstrated visible delamination and tearing of the top layer of sheet. Images of the deformed material during and after experiments showed that this behavior would ultimately cause the interior sheets to pull out of the grip region (see Fig. 7) while the outer layers remained adhered to the grips. As a result, the sheet pull-out behavior would allow the specimen to remain somewhat load bearing throughout high levels of strain and is the cause for the high measured strains-to-failure when using the grip displacement method to measure strain (upwards of 173 %). These observations motivated the development of a new spool-grip fixture to alleviate these problems and produce a more uniform stress distribution through the thickness of the specimens.

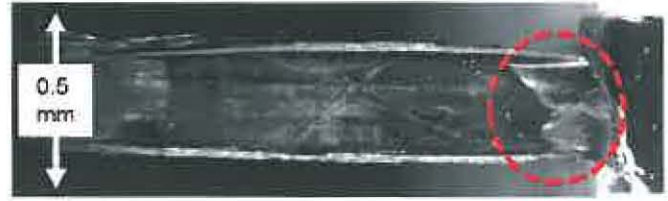


Fig. 7: Delamination observed on the outer surface of CNT sheets during adhesive-grip tension testing

Fig. 8 shows the uniaxial tension behavior of the CNT sheets using the spool-grip method. Using this gripping method, premature delamination was prevented and the sheets exhibited a 72% increase in measured tensile strength to 35.7 ± 0.5 MPa. Failure strains measured using the grip displacement method ranged from 48-86%, while directly measured strain using DIC exhibits a failure strain of 35%. Due to the large discrepancy in the elastic response of the sheets when using the grip displacement method to measure strain, the Young's Modulus was only computed using the DIC method resulting in a Modulus of 292 MPa.

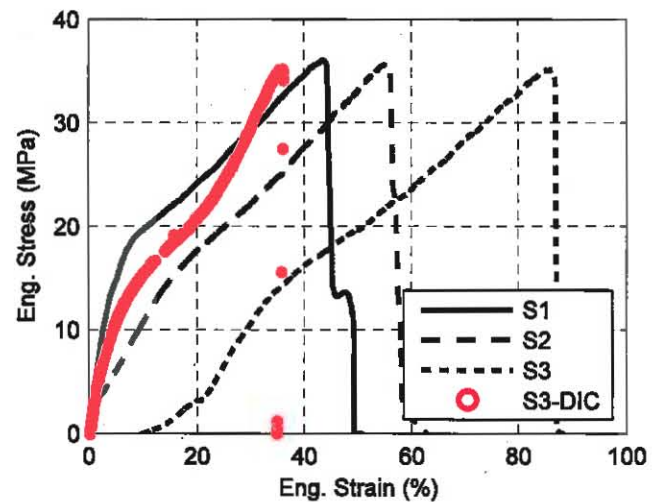


Fig. 8: Uniaxial tension test results for CNT sheets using spool-grip method

The DIC strain measurement method was found crucial for obtaining an accurate measure of specimen strain using both gripping methods. Fig. 9 compares the results between two gripping methods using DIC. Both gripping methods produce a similar elastic response up to about 20% strain. However, the specimens tested using the adhesive method experience premature failure near 21%. Using the spool grip method, premature failure is circumvented and materials are allowed to further deform up to about 36%, and as a result, demonstrate a highly non-linear elastic behavior. Furthermore, using the spool grip method the specimens are allowed to demonstrate a 72% increase in tensile strength to about 36 MPa.

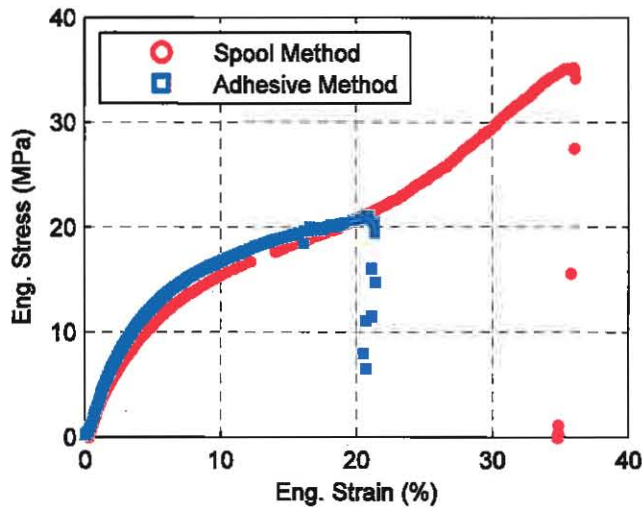


Fig. 9: Comparison of spool-grip and adhesive-grip method tension test results for CNT sheets (both using DIC)

B. CNT YARNS

CNT yarns were also tested under uniaxial tension. As shown in Fig. 10, the diameters of CNT yarns are of similar size to a strand of human hair (50 μm diameter). Due to limited quantities of CNT yarn, preliminary tension tests of strands of hair using the UV-curable adhesive grip method showed that strands would debond at the glue-specimen interface. This prompted all tension tests of CNT yarns to be conducted using the spool-grip method to circumvent the issue of debonding. Fig. 10 shows images of a CNT yarn before and after a typical uniaxial tension test.

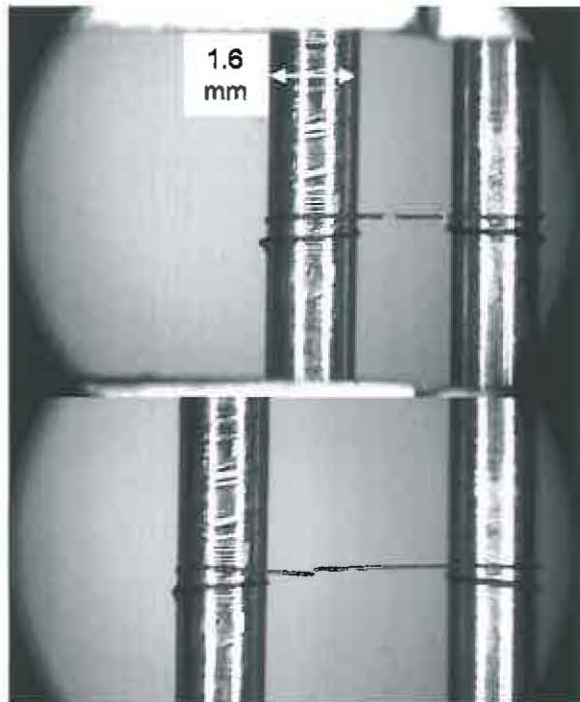


Fig. 10: CNT yarn before (top) and after (bottom) tension test

Fig. 11 shows the uniaxial tension behavior of the CNT yarns using the spool-grip method. Unfortunately, the DIC method was not successfully employed to measure strain due to insufficient speckle pattern generation on the curved surface of the yarns. Failure strains measured using the grip displacement method ranged from 30-36 %. Similar to the CNT sheets, the large discrepancy in elastic behavior when using the grip displacement method to measure strain prohibited an accurate measure of the Young's Modulus. Yarn specimens demonstrated a six-to-seven times higher tensile strength of 218-241 MPa compared to the sheet materials (35.7 MPa).

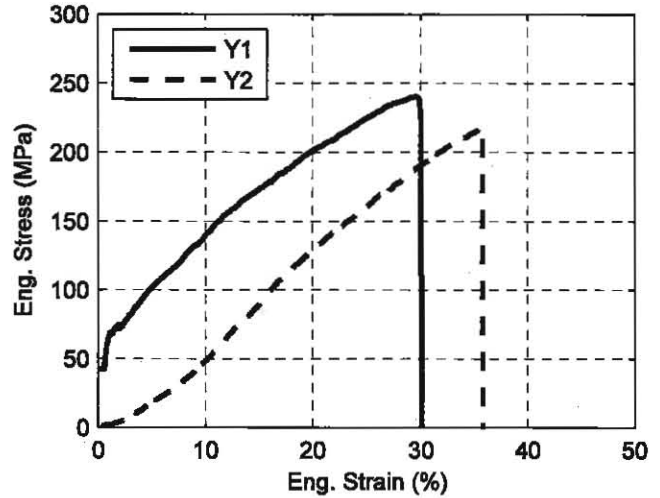


Fig. 11: Uniaxial tension test results for CNT yarns using spool-grip method

The mechanical behavior of the CNT-based materials tested in this research are summarized below in Table 1.

Table 1: Mechanical behavior of CNT-based materials

Material	Grip Method	Tensile Strength (MPa)	Young's Modulus (MPa)	Failure Strain (%)
CNT Sheet	Spool (S)	35.7 +/- 0.5	292 (DIC) N/A	35 (DIC) 43-86 (grip)
	Adhesive (A)	20.7 +/- 0.5	353 (DIC) 200 (grip)	21 (DIC) 54-173 (grip)
CNT Yarn	Spool	218-241	N/A	30-36 (grip)

DISCUSSION

In-situ microscopy of the CNT sheet materials allowed for the observation of delamination during tension testing using the initial adhesive gripping method, and failure of the sheets was attributed to the imposed loading conditions of the testing system rather than the inherent behavior of the material. It is surmised that the adhesive gripping method

produced a higher proportion of load bearing in the outer layers of the sheets due to the adhesive-sheet interface compared to interior layers. As a result, the apparently low interlaminar shear strength of the sheet materials allowed for the specimens to prematurely delaminate along the outer surface. Using the newly proposed spool grip method allows for a more uniform through-thickness stress distribution during testing, and as a result, the sheets were able to exhibit an increase in strain-to-failure by 67% and an increase in tensile strength by 72%. The spool grip method was also found to effectively measure the strength of the CNT yarn materials, whereas the use of adhesive gripping led to debonding during preliminary tests.

The measured strength of the CNT yarns tested here are similar to what has been measured for CNT yarns of similar structure and diameter with quasi-static tensile strengths of approximately 190 MPa [3]. However, the strength of the yarns was approximately six-to-seven times higher than the strength of the CNT sheet specimens. This can be attributed to the alignment of the individual CNT-fibers bundled into a rope-like structure to increase overall strength, while the non-woven sheet structure suffers from misalignment of the CNTs and remains prone to interlaminar failure due to a relatively low interfacial strength between the many randomly distributed layers of CNT material [4]. A few methods have been demonstrated to increase the strength of CNT-based materials including optimized structural-fiber design of CNT yarns with strengths as high as 1-9 GPa [5] and electron-beam radiation induced cross-linking [4]. Additional testing and analysis is suggested to analyze the interfacial strength of the CNT-based materials and further study the interfacial behavior.

Finally, it was determined that direct strain measurement is critically important for accurately characterizing the true deformation of the micro-sized specimens. Without the use of the image-based DIC method, the material might exhibit a measurably high strain-to-failure and an inconsistent elastic response. In conjunction with the spool method, it was shown that complex multi-sheet deformation behavior can be captured and artifacts related to delamination and non-uniform deformation can be circumvented. However, surface preparation to produce the required speckle pattern for DIC analysis remains a challenge and was not successful for all experimental trials. Additional work is currently being pursued to improve the effectiveness and robustness of the DIC method to measure strain for micro-sized specimens including new surface preparation techniques and three-dimensional DIC to measure strains of deforming non-planar structures.

CONCLUSIONS

Nano-based materials and structures were tested using a novel micro-scale mechanical testing system. An apparently low interlaminar shear strength leaves the CNT sheet material susceptible to delamination during uniaxial testing using the adhesive grip method. CNT sheets were observed to delaminate as a result of a higher proportion of load

bearing in the outer sheets versus internal sheets. In response to this, a new spool-grip method was used to alleviate non-uniform through-thickness stresses, circumvent premature delamination, and allow the sheet material to sustain a 67% increase in failure strain and 72% increase in tensile strength. Furthermore, tension tests of CNT yarns showed that the yarn-structure was approximately 7 times stronger than the sheet structure, owing to a higher degree of CNT alignment in the test direction.

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